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ABSTRACT

This report is in response to complaints and criticism by the library community on the Superintendent of Documents' decision to furnish third generation diazo microfiche for the Depository Library program. It reviews some of the basic photographic chemistry of both silver halides and diazos which have an influence on dark stability. A review of the literature on the subject is presented and evaluated. It demonstrates that the fears of librarians are unfounded and that furnishing all silver halide fiche would not be cost effective since mechanical stresses encountered in handling the film could well cause it to deteriorate much faster than any density changes occurring in storage. It concludes that the decision to furnish diazo fiche is eminently sound, saving the government approximately $500,000 the first year. (Author)

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ARCHIVAL STABILITY OF MICROFILM

by

Albert R. Materazzi

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ARCHIVAL STABILITY OF MICROFILM

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April 7, 1978

Summary

This report is in response to complaints and criticism by the library community on the Superintendent of Documents' decision to furnish third generation diazo microfiche for the Depository Library program. It reviews some of the basic photographic chemistry of both silver halides and diazos which have an influence on dark stability. A review of the literature on the subject is presented and evaluated. It demonstrates that the fears of librarians are unfounded and that furnishing all silver halide fiche would not be cost effective since mechanical stresses encountered in handling the film could well cause it to deteriorate much faster than any density changes occurring in storage. It concludes that the decision to furnish diazo fiche is eminently sound saving the government approximately $500,000 the first year.

Introduction

A detailed study of factors affecting the stability of microfilm has been conducted. The need for this was generated by the reaction of the library community to the decision to furnish second generation archival "quality" silver halide microfiche to regional depository libraries and third generation diazo microfiche to both regional and "selective" libraries. This reaction was, in large measure, sparked by a memorandum prepared by Mr. Jeffrey Heyman, Chairman of the RLMS Standards Committee. In this document Mr. Heyman reviewed the reasons advanced by Mr. John D. Livsey, Director, Library & Statutory Distribution Service, which led to the Superintendent of Documents' decision. He then attacked these reasons point by point by citing various authorities. The copy available to us did not include the bibliography of documents and authorities to which Mr. Heyman referred. However, we have conducted our own study of the literature and we believe that we have turned up the most pertinent. The most important papers studied and the most scientific were authored by Dr. P. Z. Adelstein and associates. The most recent appeared in Vol. 3, No. 5, Summer 1977 issue of the Journal of Applied Photographic Engineering (copy attached). We have been informed that it has been reprinted in various journals of the library community but we have not seen these reprints. If it was not reproduced in its entirety but simply digested, this could account for some of the confusion. Dr. Adelstein headed a special Task Group on the Stability of Processed Diazo and Vesicular Films established by the American National Standards Institute and had as its objectives to obtain information on the dark storage life of diazo

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processed film and to establish an accelerated aging test ("meaningful incubation test condition") which will predict the life of diazo images and which would be incorporated in an ANSI diazo film specification. This report will take a critical look at Dr. Adelstein's work later on, but it can be stated at this point that it has been widely misinterpreted by librarians. The material it contains is not easy for the average layman to understand. Before detailing our reasons for this conclusion it might be useful to briefly outline the mechanisms involved in silver halide and diazo chemistry.

Silver Halide Films

A suitably transparent plastic film, usually cellulose esters (e.g., acetate) or polyesters (e.g., polyterphthalates trademarked "Estar" or "Cronar") are coated on one side with a gelatin emulsion of silver halide (bromides, iodides or chlorides). The emulsion is specially ripened (aged) so that after exposure and processing it will produce extremely fine grains of metallic silver, these are necessary so as to obtain good resolution. The emulsion can also contain sensitizing dyes since silver halide is normally only sensitive to the ultraviolet and blue components of the spectrum. The reverse side of the film is coated with gelatin containing an anti-halation dye which prevents actinic light (i.e., light which will have a photographic effect) from being reflected back into the photosensitive layer, the gelatin also prevents the film from curling as would happen if only one side was coated.

Upon exposure to suitable light a few molecules of the silver halide salt are reduced to metallic silver and free halogen. In a closed system the action is reversible and indeed when extreme amounts of light are used silver halide will solarize, i.e., additional exposure will reduce the number of metallic silver particles formed.

The effect of the light can be multiplied many times by immersing the film in a bath containing a reducing agent (developer). This will selectively reduce the silver in situ wherever the exposure formed free silver. The developing bath contains, in addition to reducing agents, mild alkalis to swell the gelatin and permit the solution to diffuse uniformly throughout the layer. It also contains restrainers to prevent the unexposed portions from being affected. When the optimum amount of silver in the image area has been reduced the action of the developer is neutralized in a weakly acid stop bath and then the image is "fixed" by immersing it in a solution containing a chemical which will dissolve the silver halide, e.g., "hypo" (sodium thiosulfate). The fixing bath also contains materials which will harden the swollen gelatin and prevent it from being scratched or otherwise damaged. The film is then washed until most of the chemicals have been removed. The diffusion out of gelatin by washing is exponential. For example, if 90% of the salt is washed out in 5 minutes then 10% still remains, another 5 minute wash will remove 90% of these leaving a residual of 1% and so forth. Theoretically the salt concentration will never totally disappear (from p. 138 - Photo Systems for Engineers - SPSE 1966).

Standards and tests have been established by ANSI for determining maximum amounts of thiosulfate which can remain in films destined for archival storage. One source told us that some of the latest research indicates that very small amounts of thiosulfate can be beneficial and consideration is being given to amending the standards so as to include minimum amounts.

Normally processed first generation silver halide films are non-polar (negative), i.e., light portions on the original are dark on the film and vice versa. Silver halide second generation films would be positive but there is an image definition loss of about 20%. Third generation films made from this positive would result in negatives again with a 20% loss. However, it is possible to process silver halide films by reversal chemistry in which case they would be polar (i.e., positive from positive or negative from negative). In reversal processing the film is not fixed after development but instead immersed in a bleach which converts the metallic silver to a complex which is not light sensitive. The film is then re-exposed and redeveloped forming metallic silver in the image areas. It is then fixed and washed. Reversal processing does result in some loss of contrast.

If properly processed and stored silver images will last a long time. If all of the chemicals are not removed, the metallic silver will react with them and fade badly, an example of this has been experienced by all of us, particularly quick processed photographic prints. Silver is also a reactive metal and combines easily with atmospheric contaminants such as halides, sulfides, hydrogen peroxide, sulfur dioxide, etc. If film has been improperly fixed, i.e., not all of the non-image halide has been removed, such areas darken upon exposure to light and loss of contrast results. Gelatin is subject to bioattack from bacteria, molds and vermin, particularly under conditions of high heat and humidity. The hardness of gelatin is a function of temperature, it softens and will melt at about 90° C., consequently when handled at elevated temperatures it will scratch easily damaging the image. Even at room temperatures this can occur. Fingerprints must be removed immediately, otherwise the salts they contain will react with the silver.

The ultimate in stability of processed films requires as a prerequisite recommended storage conditions. Recognizing this NARS has just published proposed standards for archival storage of silver halide microfiche (p. 12734, Vol. 43, No. 59, March 27, 1978 issue of the Federal Register). A most important provision of these proposed rules is that the master fiche can only be used for duplication. Archival fiche must be stored in environmental conditions where the relative humidity will be in a range of 20 to 40 percent and changes shall not exceed more than 5% in a 24 hour period. Dehumidifiers using dessicants will not be used. The temperature will not exceed 21° C. (70° F.) and rapid, wide-range changes will be avoided, not to exceed 5% change in a 24 hour period. The ambient air will be filtered to remove dust particles which can abrade the image. Gaseous impurities shall be removed by suitable washers and scrubbers. What NARS is saying is that it is not enough to process silver halide films to archival quality, they must be stored under archival conditions.

Diazo Chemistry

The photosensitive system in diazo systems is completely different, a variety of chemicals are lumped under the term "diazo," as can be expected not all of them undergo similar reactions. Any substance which contains the diazo group (-N=N-) in its molecule is properly called a diazo. These materials are very unstable, if not properly stabilized they can explode violently. However, they can be stabilized by forming salts with metal chlorides (e.g., zinc or cadmium), fluorboric acid, etc. Most of them have a yellow color but this can be modified by adding color forming groups to the molecule. It is in this form that they are used in diazo films. During exposure the diazo nitrogen will split off leaving a colorless material behind. Like most light-sensitive compounds they are sensitive only to light in the blue and near UV.

Diazo compounds will "couple" with a variety of organic chemicals to form highly colored azo dyes which have been in use in the textile industry for years. Azo dyes are also adsorbed on inert materials to form pigments used in paints and printing inks.

Diazo films to be used in microfiche contain a suitably stabilized diazonium salt, at least two coupling agents which will form a dark composite. The mixture is coated on the film in a resin which forms a tough film when dried. In light struck areas the diazo component is decomposed and the decomposition product will no longer couple. The coupling reaction takes place best under alkaline conditions so after exposure the film is exposed to ammonia fumes and it is simultaneously "developed" (image is colored) and "fixed" (no longer light sensitive).

If the exposure has not been sufficient to destroy all of the diazo in the non-image portions that which remains will darken in time reducing the overall contrast.

Being organic azo dyes can be expected to fade, even in the dark. Particularly if they have not been properly stabilized. In addition the azo dyes comprising the image are far more insensitive to light than the original diazo compound. All colored materials absorb certain portions of incident light and over long period will fade as a result. The shorter wavelengths have the most energy, in many cases stability is increased by protecting the material from UV rich sources. Heat can also decompose the dyes but the chemical reactions are different for heat and light. In his book, "Light Sensitive Systems," Dr. Jaromir Kosar states, "In this connection, it is of interest to note that the stability of diazo compounds does not always go hand in hand with their light sensitivity, in other words, there is no direct relationship between light sensitivity and thermal stability." This is important to remember when Dr. Adelstein's paper is reviewed further on.

The photographic speed of diazo materials is directly dependent on their concentration in the coating. Therefore real dark images can only be had at some loss of speed. Generally the darker the image the longer it will take for a diazo image to fade to the point of unusability.

It should be noted that the light decomposition of organic dyes follows the laws of photochemistry. Most importantly for light to have an effect it must be absorbed. Thus red light illuminating a red substance cannot fade it since it would all be reflected or transmitted. Secondly, as light proceeds through a transparent colored material it is attenuated, thus only the molecules nearest the light source get the full amount of the energy contained in the incident light.

Diazo films have several important advantages. They can be dry processed in line with the exposing light, they cost much less than silver films, they have higher resolving power than silver permitting more faithful duplicates to be made. They are direct-positive, i.e., a positive results from a positive or a negative from a negative. Thus the original silver negative need only be used once to form a diazo duplicate from which any number of other duplicates can be formed. There is a 20% loss in image fidelity when copies are made on silver halide films as against only 4% when diazos are used. Diazo images are nearly grainless permitting larger reproductions without grain interference. The tougher resin coating will withstand more handling without damage then gelatin and it is not subject to biological attack. Because of dry processing the film can be mounted in aperture cards prior to exposure. Finally, "the diazotype image, when properly processed and stored, is contrary to general opinion, quite stable and will last for more than 50 years, which is longer than is required by most applications." (Kosar, p. 307)

Basic Sensitometry

Photographic exposure is defined by the equation

$$E = I\,T \qquad (1)$$

where I is the intensity of the light reaching the photosensitive material measured in watts and T is the time, measured in seconds, that the light is allowed to react. This is only true within broad limits. Most photosensitive materials demonstrate a reciprocity law failure. Some more than others. From equation (1) the photographic effect of 1000 watts exposing for one second should equal that of 1 watt exposing for 1000 seconds but in actual practice this is not true. Secondly, there is an intermittency effect.

Again from equation (1) 100 exposures of 1 second each should yield the same effect as 1 exposure of 100 seconds assuming equal intensity of light in both cases. And again this is not the case in actual practice.

Because of these two effects the use of constant intense light to simulate in an accelerated manner small discrete exposures to normal light is not always valid. Several librarians have reported "fading" after exposure of diazo microfiche to "printing" lights for long periods. Lights used in printers are very intense and rich in ultraviolet because they are designed to expose diazo coatings. It is not valid to equate their effect on the azo dye image in the finished fiche to that of numerous small exposures to lights used in readers which contain a much smaller ultraviolet component than do printing lamps.

The effects of exposure are studied by plotting the density against the logarithm of the exposure. Characteristic S shaped curves result from which many photographic properties can be deduced.

Density is defined by the equation

$$D = \log \frac{1}{T}$$

where T is the percent transmission of the incident light. Thus a photographic density of 1 means that the film is passing 10% of the light, 2 equals 1% transmission and 3 equals 0.1% transmission. It is evident that at the higher densities contrast is greatly reduced. For this reason the apparent contrast at the dark end of a grey scale is less than at the light end.

Density is measured under two conditions identified as visual density and printing density. The term visual is used to indicate that the receiver of the transmitted flux is, or has, a spectral sensitivity equal to that of the eye. Similarly, the term printing is used to indicate that the receiver of the transmitted flux is, or has, a spectral sensitivity equal to that of a photographic printing material. In both cases, the spectral energy distribution of the incident flux is that of a tungsten lamp operated at a color temperature of 3000° K.

Studies on light fading of diazo materials

With the above as background we can now proceed to evaluate Dr. Adelstein's work but some reference should be made to previous studies of image stability. Not much work has been reported in the literature. In 1964 Barba and McKinney studied the action of light from a 300 watt tungsten lamp on film in a reader and on the same film exposed for 8 hours to a carbon arc in a Fadeometer. They also studied accelerated aging of the material by conditioning it at 100° C. (212° F.). They measured loss of density (both visual and printing) and found varying degrees of fading with two diazo microfilms showing "superior resistance to fade." They also found much less density change after heat aging than with light exposure and concluded "dark storage should not significantly affect azo or microfilm permanence." (Paper #28 presented at SPSE Symposium on Unconventional Photographic Systems, October 1964).

Mr. Heyman and several librarians have refefred to a British report by R. N. Broadhurst on "An Investigation of the Effects of Exposure to Light

on Diazo Microfilm." We have not been able to obtain a copy. Dr. Adelstein has this to say about it: "This work has been confined to light fading studies and showed wide differences between films and a marked influence of the type of illumination." This is to be expected and we suspect that Mr. Heyman is only quoting the worst cases.

In an address to the 1973 Annual Conference of the National Micrographics Association, Dr. A. H. Stuart of Bruning Division, Addressograph-Multigraph Corporation, had this to say: "Because of the easy processing and low costs, it is quite feasible to make diazo microfilm copies for limited time use. However, diazo prints are also capable of retaining their utility over periods of prolonged storage. How long? We don't really know - reliable predictions of longevity are difficult. Fortunately, there is by this time considerable information from actual experience. Engineering films which have been stored in ordinary office files for more than 30 years can be verified from first hand experience as intact and fully usable. As mentioned above, diazo microfilms were put into use more than 25 years ago and prints which have been filed since 1945 to 1947 are reported to be in excellent condition."

At the same Conference, Dr. Joseph H. Shepard of 3M's Microfilm Division, reported on a then new microfilm. Regarding its stability he said, "... As with any diazonium material, the image is an organic dye and it is subject to fading when exposed for prolonged periods of time to light. For example, after three hours of continuous exposure in the film plane of a typical engineering drawing reader-printer, the dye image of Type 788 will fade approximately 12 to 15%. Even after this severe test, all of the information is still legible and readable prints can be produced."

In a paper presented to the 1967 Symposium on Unconventional Photographic Systems, Dr. David P. Habib stated, "In general, the file stability of properly formulated diazotype prints is excellent. Light stability, however, can vary depending upon the molecular structure of the azo dyes that comprise the image, the matrix composition in which the image is contained, and the formula components employed."

Later on he reported: "During the period of the last 40 years, there has been ample proof that suitably formulated diazotype prints are long lived. Several years ago the NABDC embarked on a program to collect information on this subject. Diazotype manufacturers were asked to supply the oldest prints available illustrating the print aging qualities of diazo materials. These have been incorporated into a book, now in the files of the Executive Secretary of the NABDC.

In addition, a detailed questionnaire was sent to the earliest users of diazo microfilm, namely the Social Security Administration and the Bureau of Public Debt, asking for their experience with diazo film. The reports were highly favorable, indicating that diazofilm showed no signs of significant deterioration over a use period of about 20 years. The NABDC Technical Practice and Standardization Committee currently is collecting data and developing test methods by which diazo microfilm can be qualified for permanent record or archival storage."

Dr. Adelstein's Studies

All but one of the studies previously reported used light to simulate fading in an accelerated mode. The Barba and McKinney study found that heat was not as effective as light. The heat mthod they used was a suggested National Bureau of Standards test. Nonetheless Dr. Adelstein chose to use heat instead of light. The rationale being that heat accelerates chemical reactions, in basic chemistry we were taught a rule of thumb that each 10° C. rise in temperature doubles the speed of the reaction. In truth rates of reaction are studied in chemical kinetics and are very complex. However, Dr. Adelstein and associates did develop a test method which is being balloted on by the ANSI Task Group. He has told me that he expects no opposition to the proposed test methods and expects them to be issued by ANSI in the summer of this year.

The standards will be used to test for "long term film" which must have a "useful life" of 100 years, to meet this standard films must successfully pass established criteria after 50 days incubation at 80° C. (176° F.). "Medium term film" must have a "useful life" of 10 years and withstand incubation for 14 days at 70° C. (158° F.). A third category is "Archival Film" whose image keeping qualities must equal those of archival silver halide.

This latter designation is purely arbitrary and is based on specifications of the Archivist of the U.S. and the ANSI Task Group and infers archival storage. As has been pointed out previously and is stressed by Dr. Adelstein, "... archival results will only be achieved if archival film is stored under archival conditions." Mr. Heyman considers this argument "not persuasive." Nonetheless it is not reasonable to use data developed for "archival conditions" and not apply such conditions equally to diazo and silver. The point is that fiche used in research libraries is subject to in use stresses which will affect its longevity much more than any changes in density will and this applies to silver as well as diazo. There is strong evidence that "useful life" can be shortened by handling the film much quicker than by light induced fading. We believe that film used for research purposes is going to have to be replaced periodically more from handling than from fading of the image. Silver halide films are more susceptible to this type damage than are diazos. If one assumes that "reasonably used" fiche have to be replaced, for whatever reason, every five or ten years, it makes no sense to pay three times as much for silver because of the chance that diazo would fade in 100 years.

There are many weaknesses in the methods used to arrive at the standards proposed by Dr. Adelstein to which he readily admits. In fact he carefully points them out in his latest report. The standards developed may be useful as quality attributes for procurement purposes but they will not absolutely predict the dark storage life of diazo film. Dr. Adelstein compares density changes occurring under various temperatures with that of silver halide which stored archivally is the standard for comparison. This is a most important distinction, one could for instance postulate storage conditions (e.g., tropical storage under ambient conditions) where silver films would not store as well as diazo films. Consequently Dr. Adelstein finds nothing wrong with the decision to furnish diazo in the Depository Library program feeling that it is eminently sound.

The first weak point which must be understood is the Task Group's definition of "usability." By their definition two changes in density are used. Such usability "applies only to commonly used standard photographic or viewing techniques and does not cover unusual reproduction approaches" (emphasis added). The first is that the density difference on the film between low and high density patches of 0.1 and 1.2 respectively must not decrease to a value of 0.8 or lower. The second criteria is that the film is considered to be no longer usable when the low density patch exceeds a value of 0.4. This applies to both visual and printing density. Thus a film fails to meet the Task Group's usability criteria when any one of four readings fails to meet the established values. The arbitrariness of this must be evident. For the low density patch to increase up to 0.4 in properly exposed and processed film infers that the reaction products of the diazo are light sensitive, not very likely from the chemistry of the reaction. In fact in the data presented by Dr. Adelstein only three out of five films showed enough change in this patch to estimate useful life and then only for printing density (none for visual density), the "useful life" prediction for these three films were 65, 350 and 600 years. Only one of these would not pass their test if only this criteria were employed. The second criteria is equally arbitrary. It may be true that "normal" reproduction methods could have difficulty with images containing a density difference of less than 0.8. But it is not difficult (much less impossible) and the human eye has no difficulty with images having this degree of contrast.

The second basic weakness is the use of heat to study reactions which are induced by absorption of light energy. In Dr. Adelstein's words, "It is not to be implied that there may not be some diazo films that exist now or in the future whose temperature dependence of density change is significantly different from that shown in Table IV." Indeed some of those tested may not be temperature dependent since out of 20 possible "useful life" extrapolations only nine could be developed. For the other eleven "Insufficient density change occurred on which to estimate a useful life." In fact, the one film for which a useful life of nine years was predicted (pointed to in horror by Mr. Heyman) failed only one of the four criteria, for the other three insufficient density change occurred. We appreciate that heat was used to develop test criteria but it should be noted that the five diazos withstood elevated temperature quite well. At 50° C. (122° F.) and measuring only printing density, two began to fail at 700 days because of film degradation, one at 880 days and one at 500 days. This is not too bad since the film would probably never be exposed to this high a temperature for so long a time.

The final weakness in the paper we would like to discuss is the treatment of the data. He used a standard method in chemical kinetics called an Arrhenius plot. The proper use of this technique requires a knowledge of the chemical reaction involved, the logarithm of the reaction rate is plotted against the reciprocal of the absolute temperature. If the reaction is linear, i.e., all data points reasonably fall in a straight line, the line can then be extended beyond the data points observed to

predict the reaction further out in time. The reaction rate of the light degradation of diazos is not known. So he used the logarithm of the number of days before the film failed one of the two criteria for both visual and printing density changes (not double but quadruple jeopardy). Dr. Adelstein was aware of this problem, "It is recognized that while this approach follows that used in chemical kinetics, it is basically an empirical treatment of the data. Arrhenius plots were made for the loss of photographic information without consideration to the reactions of the diazonium compounds."

Even so only "reasonably linear plots were obtained" (in one case he had to ignore two of the five observed data points). Arrhenius plots were obtained for only one density criteria (out of a possible four) in some of the five films studied. When more than one density end point was available only the lower estimate was used. As a result he reported a spread of estimated useful life of from 9 to 140 years whereas the actual data gave a spread of 9 to 680 years.

More could be said about the treatment of the data but it would belabor the point that we cannot base a decision on whether to use silver or diazo on this one paper. Particularly since the overall difference between diazo and silver halide fiche for the total program is $500,000. We may, however, want to use the standard, once it issues, to procure "long term film" (one hundred years "useful life"). At least two of the five films studied by Dr. Adelstin meet the criteria required for this type of film.

Other Microfilm Systems and Future Technology

There are other microfilm systems on the market or very near to coming on the market which should be mentioned. Some of these offer excellent dark storage potential and less cost than silver. Consideration should be given to incorporating them in future contracts as more information is developed. Some have potential storage properties superior to those of silver or diazo.

Vesicular Films

Besides silver halide and diazo sensitized films the best known are vesicular. In this type of film a diazo component is incorporated in a plastic (polymer) material and coated on a film base. As noted earlier when exposed to light diazos decompose liberating nitrogen gas. Normally this would diffuse out of the plastic but immediately after light exposure the nitrogen is still trapped in the plastic. Heating the film at this point causes the gas to expand forming a small bubble or vesicle. Thus an image is created composed of these very small bubbles. The image is fixed by re-exposing the film to light to destroy the diazo in the non-exposed areas.

The first films of this type made employed vinyldene chloride for the plastic coating. In storage this reacted with residual diazo decomposition products liberating hydrogen chloride, a very corrosive material. For this reason vesicular films were suspect for a long time.

This deficiency has been corrected and no less an authority than Mr. Peter Scott from MIT believes that they have shelf life stability superior to that of either silver or diazo.

Since 1964 the stability of processed microfilm images has been the subject of several studies. Properly stored and processed they have excellent keeping qualities. Moreover, practical tests have demonstrated excellent storage stability under semitropical ambient conditions.

There are several thermally developed diazonium and dry silver microfilm products on the market. We have not had an opportunity to study them in any detail.

"Add-on" films consist of either an organic or inorganic semiconductor coated on a suitable film base. These can be electrostatically charged, exposed and toned in the usual xerographic method. Scott Graphics and Microx already market such films. George Hodgins of Scott Graphics has told me that the dark storage properties of their TEP film is excellent, he has promised to furnish data to us. Another film which has been discussed recently is the Ovionic method being marketed by 3M.

Theoretically Coulter Information Systems' "K-C" film should have outstanding storage properties. It consists of cadmium sulfide crystals coated on a polyester film base. It is charged, exposed and toned using a liquid toner. The image is then fused. All of the ingredients of this system are extremely stable even under accelerated aging conditions.

Conclusion

Nothing developed in the course of this study justifies the additional $500,000 expenditure should we convert to all silver microfiche in the Depository Library Program.